Rapid Determination of Fat in Meat and Meat Products by Foss-let Solvent Extraction and Density Measurement

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The commercially available Foss-let fat analyzer was evaluated for the determination of fat in meat and meat products by comparison with AOAC method 24.005(a). With the Foss-let procedure, mechanical and instrumental equipment is used to determine fat in 7–10 min. A sample is extracted with tetrachloroethylene in a mechanical orbital shaker for 2 min and the specific gravity of the extract is measured in a magnetic float cell controlled by a digital potentiometer. During extraction, anhydrous calcium sulfate absorbs moisture droplets originating from the sample. The variations of comparative determinations on 67 meat samples containing 1.1–95.4% fat and 17 frankfurter samples containing 17.3–37.3% fat were analyzed statistically by grouping the data according to meat type (beef or pork) or frankfurters and into 6 ranges of fat content, and by treating the entire set of data. Error analysis of the differences and standard deviation of each grouping of paired determinations by the Foss-let and AOAC methods indicated that meat type and fat content >7.5% were not significant ($P = 0.05$) sources of variation as determined by *t*-tests on the statistics from the blocks of data. Determinations on samples containing ≤7.5% fat were consistently low and an additive correction of 0.25% was indicated. From the overall results, the accuracy and precision of the method were characterized as follows: the mean Foss-let method determination was high by 0.08% fat relative to that by the AOAC method; repeatability of ±0.31% fat between duplicate determinations compared favorably with ±0.38% obtained with the AOAC method; and precision between paired determinations by the 2 methods was ±0.44%. Both a *t*-test for significance ($P = 0.05$) and the linear regression of the 84 comparative determinations indicated that the Foss-let method was equivalent to the AOAC method for determining fat.

Pettinati *et al.* (1, 2) recently published a review and preliminary evaluation of rapid methods for determining the fat content of meat and meat products. Evaluations were based mainly on potential savings in time as compared with the official AOAC method 24.005(a) (3) and information on accuracy and precision, which frequently was minimal. The Foss-let fat analyzer method was among the more promising of those selected for detailed study.

Foss-let fat analyzing equipment (Foss America, Inc., Fishkill, NY 12524) is described in considerable detail in the published evaluations cited below concerned with its use for determining fat in meat and meat products. Essentially, the equipment embodies a new application of principles for extraction and specific gravity measurement by providing rapid and convenient extraction of fat by means of a mechanical orbital shaker and instrumental measurement of the tetrachloroethylene extracts with a magnetic float cell.

Pfeiffer *et al.* (4) compared results with the Foss-let and Soxhlet extraction methods for pork and beef samples containing 15.3–20.4% fat. They reported that results with the Foss-let method indicated the repeatability was ±0.30% fat, the accuracy relative to Soxhlet method determinations was equivalent, and the standard deviation of the differences between paired means was ±0.25%. They also obtained good correlation by linear regression of determinations with the Foss-let method and a refractometric method from 160 determinations of meat and meat products. Usher *et al.* (5) evaluated the Foss-let method for a variety of food products, including 20 samples of meat and meat products containing 5.3–41.1% fat, by comparing results with those by ether extraction which was performed following either hydrolysis of sample with hydrochloric acid or drying of sample by admixture with calcium sulfate. The mean Foss-let method determination value of 12 of the samples was 0.5% low relative to that by the hydrochloric acid-ether extraction method and the mean of 8 of the samples was 0.26%

high relative to that by the calcium sulfate-ether extraction method. The overall mean of the determinations by the Foss-let method was 0.08% high and the standard deviation of the differences between paired determinations by the compared methods was ±1.2%. Egberg *et al.* (6) also evaluated the Foss-let method for various food products, including 20 samples of pork and 16 samples of ham-bacon blend containing 28.1–54.2% fat, as determined by comparative analysis with AOAC method **14.019** (3) (hydrochloric acid hydrolysis followed by Mojonnier-type extraction). The repeatability of the Foss-let determinations was ±0.45% fat and the standard deviation of the differences between paired determinations by the 2 methods was ±1.4%. The mean difference they obtained from comparative results for each product category was not cited but reportedly was used to establish how much additive correction to apply to Foss-let determinations of each type of meat product.

Eslami-Matin *et al.* (7) compared results of fat determined in meat products by use of the Foss-let, Soxhlet, and 3 other methods. From the comparison of single determinations by the Foss-let method and duplicates by the Soxhlet method, values for frankfurter containing 21.8–37.8% fat were 0.45% low relative to the Soxhlet method and the standard deviation of the differences between the paired determinations (calculated from the published data) was ±0.49%; values for liverwurst (35.7–51.7% fat) and dry sausage (25.0–51.6% fat) were both 0.60% high relative to the Soxhlet method and the standard deviations of the differences were ±0.52 and ±0.44%, respectively. In a separate experiment with the 3 products, the overall reproducibility of the Foss-let method was determined to be ±0.24% fat.

The evaluations of Foss-let method accuracy and precision cited above indicate that the method merits consideration for status as an AOAC method. The speed of the Foss-let analyzer (a fat determination in 7–10 min) is attractive and, when combined with the other characteristics of reasonable cost, simplicity of operation, and low hazard for the operator, the method is potentially useful for meat analysts. The present critical study of the performance characteristics of the Foss-let method was made to establish optimally effective procedures for applying the Foss-let instrument, to broaden the variation in sample composition, and to apply thorough statistical treatment of the data in order to provide a basis for the proposed multilaboratory AOAC study.

METHOD

Principle

Rapid extraction of fat by a solvent, tetrachloroethylene, is provided by the strong mechanical action of a motor-driven, orbital shaker device. A weighed portion of sample, a measured volume of solvent, and a known weight of an anhydrous salt to absorb moisture droplets originating from the sample are placed in a stainless steel cup with press-on cover sealed by an O-ring. The cup assembly is fastened into the shaker and the mixture is shaken 2 min to produce an extract. The contents of the cup are rapidly filtered under pressure applied by a device which is part of the equipment. The filtrate flows directly into a chamber in which the specific gravity of the extract is measured. The chamber is thermostatically maintained at 37°C and contains a miniature hydrometer at the base of which is a small bar magnet. By increasing the strength of a magnetic field surrounding the chamber, the magnetic force eventually causes the hydrometer to rise to the top of the chamber. To accomplish this, a multiturn digital potentiometer is rotated until the hydrometer rises and the digital reading is recorded. Fat content is determined from this reading by using a chart with the calibration of the potentiometer in terms of fat content.

Apparatus

(a) *Foss-let fat analyzer.*—Includes orbital shaker, specific gravity readout unit, solvent dispenser, reference standard oil (for periodic check of potentiometer calibration), stainless steel cup with cover and 8 mm bore brass hammer, pressure filtration device, and conversion chart (Foss America, Inc., Rte 82, Fishkill, NY 12524).

(b) *Drying agent.*—Plaster of Paris (Red Top Brand, U.S. Gypsum Co., or equivalent, distributed locally through paint, hardware, or building supply dealers), 8 mesh Drierite (J. T. Baker Chemical Co., Philipsburg, NJ 08865, No. L056), or anhydrous calcium sulfate (Baker No. 1458).

(c) *Tetrachloroethylene.*—Technical grade (distributed locally through dry cleaning suppliers or Fisher Scientific Co., 191 S. Gulph Rd., King of Prussia, PA 19406, No. C-182).

Determination

Prepare samples for analysis according to **24.001**. Check calibration of Foss-let potentiometer each

day by using solvent to set zero point and reference standard oil to set 50% fat point. Using either top-load or triple-beam balance with 0.1 g sensitivity, tare Foss-let cup after setting brass hammer on its spindle. For products containing $\leq$60% fat, weigh 45.0 g sample into cup; for products containing >60%, weigh 22.5 g. Add ca 80 g Plaster of Paris (or ca 60 g anhydrous calcium sulfate). Dispense 120 ml tetrachloroethylene into cup. Press cover onto cup and install in orbital shaker. Set shaker timer for 2 min and turn unit on. While extraction proceeds, assemble pressure filtration device by first placing 7 cm D circle of Whatman No. 50 and then 7 cm D circle of Whatman phase-separating paper (1 PS) into perforated base. After 2 min extraction, remove cup from shaker, lift cover, and remove brass hammer from cup. Immerse cup in ice-water bath ca 0.4 min while stirring with thermometer to cool contents from 47–52°C to ca 40°C. Wipe water from outer surface of cup and pour contents into assembled filter. Place piston at top of filtration device and slowly press extract through measuring system. Depress drain valve button when extract appears in overflow tube and let chamber drain; then release valve button. Repeat filling and draining 2 more times until 40–50 ml extract have flowed through, retaining final 10 ml extract in measuring chamber. Remove filtration device, slide viewing lens into position, rotate control of read-out potentiometer clockwise until hydrometer rises, and record reading. Establish that extract is at chamber temperature by repeating reading 3–4 times. Average readings and convert into per cent fat by means of conversion chart (multiply per cent fat from chart by 2 if 22.5 g portion of high-fat sample was taken).

Results and Discussion

Fat determinations on 48 beef, 19 pork, and 17 frankfurter samples are shown in Tables 1–3. The meat samples, prepared by mixing lean and fatty tissues, provided beef containing 1.1–95.4% fat and pork containing 4.6–79.4% fat. A few samples of individual muscles and fatty tissues were ground and analyzed as such. For example, the last 3 determinations listed in Table 1 and the last one in Table 2 were obtained on samples of fatty tissue without added lean. The lean-to-fat ratio was controlled in processing frankfurters so that samples were obtained with fat contents ranging from 17.3 to 37.3%.

Error analysis of the comparative determinations was performed according to the procedures suggested by Youden (8). All determinations were performed in duplicate so that precision of repeatability of the 2 methods could be determined and compared. The comparative results were also treated by statistical data reduction methods to appraise the accuracy and precision of the method relative to determinations by the official method. By paired variate analysis (8, p. 28), an overall comparison of data obtained with the 2 methods was made and the significance of type of meat or product and fat level as experimental variables was determined from the respective groups of data. By regression analysis (8, p. 40), the linear relationship and degree of association, or correlation, of determinations by the 2 methods were obtained. The standard deviations that were calculated are reported as $\pm$ per cent fat and each value is an

Table 1. Comparative duplicate determinations for per cent fat in 48 samples of beef by the Foss-let and the AOAC methods

Foss-let	AOAC	Foss-let	AOAC	Foss-let	AOAC
0.97,0.97	1.01,1.08	10.68,10.67	10.69,10.82	20.82,20.93	19.64,19.79
1.34,1.51	1.66,1.71	11.62,11.67	12.27,12.43	21.26,21.52	21.04,20.98
1.59,1.28	1.89,1.67	12.86,13.18	12.57,12.67	22.18,21.67	21.02,21.46
1.82,1.89	2.21,1.88	12.92,13.10	12.71,13.18	22.93,22.96	22.55,22.75
3.52,3.47	3.77,3.75	14.11,13.93	13.47,13.06	25.24,24.03	24.36,24.65
5.36,5.32	5.76,5.84	13.37,13.51	13.52,13.20	26.89,26.04	26.73,25.73
6.03,5.98	6.13,6.19	14.23,13.48	14.20,14.10	30.34,29.54	30.78,30.40
7.21,7.26	7.42,7.30	14.92,14.47	14.45,15.14	30.93,31.02	30.44,30.94
7.17,6.96	7.60,7.34	15.61,15.59	15.24,15.50	33.55,33.65	33.46,33.74
7.85,8.08	8.11,7.85	15.99,15.86	16.08,15.66	35.66,35.78	35.32,34.17
8.43,8.27	7.92,8.12	17.34,15.65	15.61,16.78	35.76,35.51	35.31,35.02
7.97,8.22	8.05,8.25	18.03,18.12	18.12,18.19	41.17,40.89	41.18,40.14
8.02,7.98	8.58,8.36	19.09,19.27	18.80,18.76	46.77,46.07	45.52,47.18
9.27,8.86	8.46,8.51	19.40,19.65	19.19,18.99	78.56,79.90	79.35,78.10
8.74,8.61	8.95,8.87	18.72,19.09	18.89,19.32	91.18,91.66	90.70,91.10
8.84,8.76	9.41,9.50	19.47,19.32	19.15,19.30	97.04,96.94	95.30,95.40

Table 2. Comparative duplicate determinations for per cent fat in 19 samples of pork by the Foss-let and the AOAC methods

Foss-let	AOAC
4.83, 4.83	4.63, 4.63
6.81, 6.67	6.99, 7.79
8.51, 8.42	8.88, 8.64
12.43,12.76	11.20,12.50
13.45,13.59	13.43,13.92
15.41,16.03	16.46,15.15
15.95,15.71	15.99,16.11
18.64,18.10	17.85,18.59
19.07,18.73	18.34,19.24
23.62,22.59	22.73,22.88
32.48,32.43	32.73,32.34
40.57,41.21	41.44,40.62
41.04,41.81	41.39,41.65
43.12,43.61	43.78,43.62
45.66,46.25	45.91,45.88
47.68,47.72	48.39,47.23
49.99,50.06	50.39,49.83
57.40,57.01	57.84,57.19
78.68,78.28	79.34,79.43

Table 3. Comparative duplicate determinations for per cent fat in 17 samples of frankfurter by the Foss-let and the AOAC methods

Foss-let	AOAC
17.51,17.53	17.36,17.15
18.50,18.50	17.93,17.13
25.96,25.95	25.60,25.63
25.38,25.67	25.60,25.75
26.58,26.57	25.93,26.48
27.80,26.60	27.05,26.87
26.33,26.65	27.24,26.84
28.25,28.04	28.11,28.03
27.46,27.85	28.05,28.09
30.62,30.94	30.22,30.19
30.60,30.73	30.24,30.52
31.42,31.76	30.92,30.22
30.58,30.58	30.79,30.39
31.12,31.28	31.10,30.75
32.45,32.35	32.27,32.91
36.50,36.41	36.41,35.98
37.17,37.33	37.53,37.15

estimate of the $\pm \sigma$ variability of the particular data group.

Statistics calculated for the meat type or product groups of samples and for the combined sets of data are shown in Table 4. Repeatability of the Foss-let method, calculated from differences between duplicate determinations, was ± 0.33, ± 0.32, and $\pm 0.25\%$ fat for beef, pork, and frankfurter samples, respectively, which compared favorably with repeatability of the AOAC method. Mean difference between results indicated that the Foss-let procedure provided good accuracy, being 0.12% fat high for beef samples, 0.10% low for pork, 0.19% high for frankfurters, and 0.08% high overall relative to AOAC method determinations. From the differences between each paired result by the 2 methods, the precisions relative to the AOAC method were calculated to be $\pm 0.47\%$ fat for beef, $\pm 0.35\%$ for pork, $\pm 0.42\%$ for frankfurters, and $\pm 0.44\%$ overall. This order of precision compared very favorably with the $\pm 0.54\%$ value obtained for reproducibility between duplicate determinations by the AOAC method. The coefficient of variation, standard deviation as a percentage of the mean fat content of the samples, indicated that fat determinations by the 2 methods agreed with a relative precision of $\pm 1.8\%$ of mean (24.5%) fat content. The between-methods mean difference of each group of results and the overall mean difference, divided by its corresponding standard error, were not significant by *t*-tests (8, p. 28) at the 95% probability level, indicating that there was no significant difference between the results for fat content determined by the 2 methods for beef, pork, or frankfurters of widely varying fat content.

To determine accuracy and precision at various fat levels, the comparative results on the same beef and pork samples discussed above were grouped into 6 ranges of fat level as shown in Table 5. From the differences between paired results, there was a general trend for the mean difference to increase as a function of increasing fat level although at each of the fat levels except the lowest (1.0–7.5% fat), the mean difference was substantially less than the respective standard deviation of difference. The latter statistic varied between ± 0.2 and $\pm 0.4\%$ fat through all fat levels except the highest (78.7–94.4% fat), at which it was $\pm 1.0\%$. The reason for this may be in part due to the requirement that only half the amount of the usual sample weight is taken for a determination when fat content exceeds 60% and the conversion chart reading is multiplied by 2. The coefficient of

variation decreased from 5.2 to 0.5% through 5 levels of increasing fat content and was 1.2% at the sixth and highest level. The t-value calculated from the comparative data of each moisture level was used to test the statistical significance of the results. Of these, only the t-value for the lowest fat level exceeded the tabular value and was significant ($P = 0.05$). This indicated that determinations by the Foss-let method on 11 samples of $\leqslant$7.5% fat were consistently 0.25% low relative to AOAC method determinations, and can be additively corrected by 0.25% fat with justification. The t-tests at the fat levels >7.5% were not significant at the 95% probability level, indicating that there was no significant difference between the values determined by the 2 methods.

Correlation of all the comparative determinations shown in Tables 1–3 was calculated by linear regression to summarize the equivalence of the accuracy and precision of the 2 methods. The regression of the 84 Foss-let method determinations (Y) relative to those by the AOAC method (X) yielded the equation, $Y = 1.006X -$

Table 4. Statistical analysis of fat determinations on beef, pork, and frankfurter sample groups and overall by Foss-let and AOAC methods

		Fat, %							
		Mean[a]		Std dev. between duplicate detns		Results between methods, Foss-let − AOAC		Comparison of results, Foss-let vs. AOAC method	
Type of sample	No. of samples	Foss-let	AOAC	Foss-let	AOAC	Mean diff.	Std dev.	CV, %[b]	t-value[c]
Beef	48	20.84	20.72	0.33	0.36	0.12	0.47	2.26	1.73
Pork	19	30.29	30.39	0.32	0.48	−0.10	0.35	1.16	−1.27
Beef and pork	67	23.52	23.47	0.33	0.39	0.05	0.45	1.91	1.00
Frankfurter	17	28.50	28.31	0.25	0.32	0.19	0.42	1.48	1.89
Overall	84	24.53	24.45	0.31	0.38	0.08	0.44	1.81	1.71

[a] All samples were analyzed in duplicate by both Foss-let and AOAC methods.
[b] The coefficient of variation (CV) expresses the *relative* measure of variation and is defined as the ratio of 2 statistics, the sample standard deviation and the sample mean: CV = (100 std dev.)/$\bar{X}$.
[c] These values do not exceed tabular t-values at the 95% probability level, indicating that there was no significant difference between the values for fat content obtained by the 2 methods.

Table 5. Statistical analysis of fat determinations, grouped by fat level, of beef and pork samples by Foss-let and AOAC methods

		Fat, %						
				Results between methods, Foss-let − AOAC			Comparison of results, Foss-let vs. AOAC method	
		Mean[b]		Difference				
Fat content of sample groups,[a] %	No. of samples	Foss-let	AOAC	Range	Mean	Std dev.	CV, %[c]	t-value
1.0– 7.5	11	4.22	4.47	−0.65–0.20	−0.25	0.22	5.2	−3.65[d]
7.9– 9.5	8	8.43	8.53	−0.66–0.58	−0.10	0.41	4.9	−0.69[e]
10.7–19.8	23	15.80	15.66	−0.70–1.2	0.14	0.40	2.5	1.64[e]
21.0–35.2	12	28.24	27.98	−0.65–0.97	0.26	0.40	1.4	2.20[e]
40.6–57.5	9	46.01	46.07	−0.33–0.37	−0.06	0.21	0.5	−0.88[e]
78.7–95.4	4	86.53	86.09	−0.91–1.6	0.44	1.04	1.2	0.84[e]

[a] Grouping based on average of duplicate determinations by AOAC method.
[b] All samples were determined in duplicate by both Foss-let and AOAC methods.
[c] See footnote *b*, Table 4.
[d] This value exceeds the tabular t-value at the 95% probability level and is significant, indicating that results by the Foss-let method on 11 samples containing ≤7.5% fat, being consistently lower than by AOAC method, may require an additive correction of 0.25% fat.
[e] This value does not exceed the tabular t-value at the 95% probability level, indicating that there was no significant difference between the values for fat content obtained by the 2 methods.

0.05. A correlation coefficient, r, of 0.9997 indicated that determinations by the 2 methods were highly correlated. A coefficient of determination, r^2, of 0.9994 indicated that 99.94% of the total variation of the Foss-let determinations could be attributed to, or accounted for, by variation of the AOAC method determinations (covariance) and 0.06% to random factors. The standard deviation from regression, ± 0.44, equaled the overall standard deviation calculated by difference analysis, shown in Table 4. The standard error of the intercept, -0.05, was determined to be ± 0.08 and a t-test of this value was not significant ($P = 0.05$), which indicated that the intercept was not significantly different from zero.

Conclusions and Recommendation

The performance characteristics of the Foss-let method demonstrated that the mechanical-instrumental equipment was adequate for the rapid (7–10 min) determination of fat content in meat and meat products. The orbital shaker provided fat extraction in 2 min without the need for pre-drying a sample which, for fat contents >7.5%, was as efficient as that obtained with a 4 hr ether extraction following a 1.5 hr oven drying of the sample. The magnetic float cell with the read-out potentiometer afforded sufficient sensitivity for the specific gravity range of fat extracts to be measured. The accuracy of the Foss-let method for the rapid determination of fat in fresh meat and emulsified meat product is equivalent to that of the AOAC method for all fat levels >7.5% and determinations ≤7.5% may require an additive correction of 0.25% fat. The precision of the Foss-let method in regard to both repeatability of duplicate determinations and between-paired determinations compared favorably with the repeatability of the AOAC method. It is recommended that study be continued to evaluate the Foss-let method collaboratively for its suitability as an alternative official method.

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